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APPLICANT: Manfred Fuchs et al.

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TITLE: "METHOD FOR IMPROVING THE OPTICAL
SEPARATION OF FLUORESCENT LAYERS"

Commissioner for Patents
Box 1450
Alexandria, VA 22313-1450

S I R:

I, Charles Bullock, declare and state that I am knowledgeable in German and English, and I hereby certify that the attached translation of German Application 100 01 671.5, filed in the German Patent and Trademark Office on 17 January 2000, is truthful and accurate to the best of my knowledge.

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Siemens AG
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Priority Document concerning the Submission
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File number: 100 01 671.5

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Applicant/patent holder: Siemens Aktiengesellschaft, München/DE

Title: Method for improvement of the optical separation
of luminophore layers

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The attached pieces are a correct and precise reproduction of the original documents of this application.

München, the 19th June 2000
German Patent and Trademark Office
The President
by order
[signature]

Description

Method for improvement of the optical separation of luminophore layers

The invention relates to a method for improvement of the optical separation of needle-shaped luminophore layers which are formed by vapor deposition onto a substrate, in particular for X-ray detectors.

Needle-shaped luminophore layers can be produced in the vapor deposition of alkali halides (for example NaCl, CsI [sic], CsBr, RbBr, RbFBr, RbFCl) as absorber material for X-ray radiation or, respectively, as host lattice for the dopant substances such as, for example, NaI [sic], EuBr₂, TlI [sic], GaBr, EuCl₂ etc. The vapor deposition of luminophore materials is an alternative for the production of binderless luminophore memory panels. In order to obtain a good modulation transfer function (MTF) for these luminophore layers, small needle diameters ($< 40 \mu\text{m}$) are necessary, whereby the needles must be surrounded with cracks of 0.3 to 3 μm for optical separation. However, this requirement can be met only with very special environmental conditions during the production of the luminophore layers.

The utilization of the differences in expansion between the substrate and the vapor-deposited luminophore layer has become established as the most frequently practiced method for producing the required cracks between the needles. The luminophore layer was thereby vapor deposited at a relatively high substrate temperature, and - given the correct material selection - the luminophore layer shrinks more strongly than the substrate situated below, which leads to thermal stresses and thus to cracks. Examples of the correct choice of material are CsI [sic] on aluminum and CsBr on glass. However, there is the disadvantage with the method described that the needle diameter of the luminophore layer increases substantially with increasing substrate temperature, and therefore the MTF decreases.

A second variant for generating fissures or cracks in the luminophore layer that are necessary

for a good MTF is to carry out vapor deposition at low substrate temperatures (50°C), and to subject the layer thereafter to heat treatment (500°C). The disadvantage of this method is grounded in the recrystallization of the luminophore layer at the tempering step. A “grain coarsening” occurs in this case, thereby leading to larger block widths and thus to a poorer MTF.

A third variant is “oblique vapor deposition”. In this method, the vapor jet of the luminophore material strikes the substrate at a flat angle during vapor deposition. A good separation between the needles is achieved by “shadowing” as early as in the seeding phase. In the practical implementation, either the substrate is arranged rotating obliquely relative to the evaporator boat, or the vapor jet is “directed” to a horizontally rotating substrate in a fashion channeled by “chimneys”. The essential disadvantages are to be seen in that, firstly, the vapor deposition of large substrates ($\varnothing > 40$ cm) is very expensive in terms of apparatus and, secondly, the needles grow obliquely onto the substrate in accordance with the angle of incidence of the vapor jet. This results in different layer properties over the surface (vignetting effect in the case of X-ray excitation).

A fourth variant of the prior method of production consists in vapor deposition of the luminophore material onto a structured support (wire fabric, photoetched substrate etc.), whereby the “depressions” in the substrates represent a barrier against surface diffusion, and the “raised” surface elements exert a “shadow effect” on the “neighbors”.

In addition to the frequently very complicated method cycle in the production of the luminophore layers according to one of these known methods, they do not all lead to an optimum optical separation of the needle-shaped luminophore layers.

It is therefore the object of the invention to configure the method of the type mentioned at the beginning to the effect that upon vapor deposition there are needle structures which are of the smallest possible diameter and are optically separated from one another effectively by cracks, such that an improvement of the optical separation of the luminophore layers ensues.

In order to achieve this object, it is provided according to the invention that the vapor deposition is controlled such that the luminophore layer is deposited on the substrate with a reduced density, whereby via the reduced density (of 5% to 50%) fissures ensue in the layer between the luminophore needles and pores or, respectively, defects and dislocations ensue in the crystal structure (lattice defects).

In order to reduce the density, it is provided in a refinement of the invention that, before striking the substrate, the vapor jet is cooled, preferably by leading cool inert gas, for example argon, through the vapor-deposition apparatus.

The method is thereby configured in a further refinement of the invention such that the gas pressure of the inert gas which is introduced by a control valve into the vapor-deposition apparatus and discharged again via a pump is below 10 Pa, preferably between 1 Pa and 3 Pa.

A very considerable reduction in density results from this operation using cooled inert gas - the temperature of the inert gas is, for example, between 0°C and 100°C, preferably at approximately room temperature, whereby the vapor with a temperature of approximately 650°C can, of course, be strongly cooled - when the vapor strikes the preferably likewise cooled substrate, whose temperature is preferably kept at between 50°C and 200°C. The combination of the cooling of the vapor jet and the cooling of the substrate to a temperature that is much lower than the vapor temperature directly ensures a substantial reduction in density, and thus a good optical separation of the luminophore needles resulting therefrom.

Physically, the reduced density can be explained with larger lattice spacings in the case of phase conversions of polymorphous crystals (NaCl type or CsCl type in the case, for example, of CsBr, CsCl, TlBr etc.) and/or by the formation of lattice defects through high vapor deposition rates - in accordance with a development of the invention the vapor deposition rate is preferably above $1 \text{ mg cm}^{-2} \text{ min}^{-1}$ - and/or by the "freezing" of the lower density of the liquid. The density of CsBr, for example, is 3.05 g cm^{-3} in the liquid state, and 4.44 g cm^{-3} in the solid state. The ratio at which

the reduced density becomes noticeable either as fissures between needles or as lattice defects inside the needles can be influenced by the vapor deposition rate.

In the event of excessively high inert gas pressure and/or excessively low substrate temperature, however, no more luminophore needles can be formed because new seeds are always forming on the surface that no longer connect to the already condensed layer. Substrate temperatures of between 50°C and 200°C and argon pressures of between 1 Pa and 3 Pa, for which a 10-30% reduction in density is achieved, have proved to be virtually optimum.

Further advantages, features and details of the invention emerge from the following description of an exemplary embodiment and with the aid of the drawing, in which:

Figure 1 shows a schematic representation of a vapor-deposition apparatus for carrying out the method according to the invention, and

Figure 2 shows an REM exposure of the luminophore layer that was applied to a substrate with a substrate temperature of 160°C in conjunction with an argon pressure of 2 Pa.

The vapor-deposition apparatus, outlined schematically in figure 1, for carrying out the method according to the invention comprises a vacuum vessel 1 in which a vapor-deposition source 2 is arranged opposite the substrate 4, which preferably rotates around an axis 3. Via a control valve 5, an inert gas, such as argon, which is at a very much cooler temperature, for example room temperature, than the vapor temperature of typically 650 to 700°C, can be introduced into the vacuum vessel 1 that preferably firstly strikes a baffle 6 and is not introduced directly into the vapor jet 7. The inert gas is extracted via a vacuum pump 8, whereby the setting ensues such that a pressure of below 10 Pa, preferably between 1 Pa and 3 Pa, results inside the vacuum vessel 1. The substrate 4 is kept at a substrate temperature of between preferably 50°C and 200°C with the aid of a cooling apparatus 9 (not externally shown), whereby in conjunction with the cooling of the vapor jet 7 a reduced density of 10 to 30% of the needle-shaped luminophore layer precipitated on the

substrate ensues that in turn entails a substantial restructuring, and therefore a good optical separation of the individual needles from one another. One recognizes this structuring very well in figure 2, in which the bright needle-shaped structures of the luminophore layer are all virtually completely separated from one another by cracks of dark appearance. The dark points inside the needles are defects in the needles.

Patent Claims

1. A method for improvement of the optical separation of needle-shaped luminophore layers formed by vapor deposition onto a substrate, characterized in that the vapor deposition is controlled such that the luminophore layer is deposited on the substrate with a reduced density.
2. Method according to claim 1, characterized in that the vapor jet is cooled before striking the substrate.
3. Method according to claim 2, characterized in that cooling ensues by leading cool inert gas through the vapor-deposition apparatus.
4. Method according to claim 3, characterized in that the gas pressure of the inert gas introduced into the vapor-deposition apparatus via a control valve, and thereby preferably diverted via a baffle and directed away again via a pump, is below 10 Pa, preferably between 1 Pa and 3 Pa.
5. Method according to any of the claims 2 through 4, characterized in that the inert gas is argon.
6. Method according to any of claims 3 to 5, characterized in that the inert gas is introduced with a temperature of between 0°C and 100°C, preferably at approximately room temperature.
7. Method according to any of the claims 1 to 6, characterized in that the substrate is cooled during vapor deposition.
8. Method according to claim 7, characterized in that the substrate is kept at a temperature of between 50°C and 200°C.
9. Method according to any of the claims 1 through 8, characterized in that a vapor deposition

rate is selected that is greater than $1 \text{ mg cm}^{-2} \text{ min}^{-1}$.

Abstract

Method for improvement of the optical separation of luminophore layers

The invention relates to a method for improvement of the optical separation of needle-shaped luminophore layers formed by vapor deposition onto a substrate. The vapor deposition is thereby controlled such that the luminophore layer is deposited on the substrate with a reduced density.

Fig. 1